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MORPHOLOGICAL IDENTIFICATION OF SOME ORGANIC ACIDS AS THE SODIUM SALTS

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In an investigation of the characterization of organic acids by quantitative microdiffusion (1), it was observed that a number of the acids gave large characteristic crystals of the sodium salts as a result of the diffusion process. These crystals can serve to confirm the identity of the diffusing acid, as well as provide, by their speed of formation, a rough index of the diffusion rate. The technique may also be used to prepare large crystals of the sodium salts of the acids.

The acids diffused from appropriate solutions in the outer well of a sealed Conway cell to one or two sodium hydroxide pellets in the inner well. As expected (1), characteristic crystals formed only in those cases where diffusion was most rapid.

PROCEDURE

The nonaqueous solutions were allowed to diffuse in 7.5 cm. Conway cells (Microchemical Specialties Company, University Avenue and Grove Street, Berkeley, California), while the smaller aqueous samples were run in 3 cm. cells. One to two pellets of base were used in all cases. The mixtures contained approximately equal volumes of the acids; the total acid concentration was about 2 percent. Tight-fitting tops were sealed onto the cells with petrolatum. Diffusion times, volumes, and other details are given in Table I.

Photomicrographs were taken of significant samples by incident illumination, using a Galileo Model ZC microscope.

RESULTS AND DISCUSSION

The results are summarized in Table I. Characteristic crystal forms are shown in the figures. It will be noted that the mixture of formic, propionic, and isovaleric acids gave crystals characteristic of isovaleric acid, while the mixture of valeric and isovaleric acids produced no crystals.

Crystals of the sodium salts may be readily removed from the sodium hydroxide pellets by allowing one or two drops of chloroform to fall on the crystals. In this manner large, well-formed crystals of sodium salts may be prepared for crystallographic study.

LITERATURE CITED

(1) Marshall, L. M., and Fox, F. T., Anal. Chem. 30, 140-143 (1958).

Table I. Crystal Formation During Microdiffusion of Acids to Sodium Hydroxide

Acid	Solvent[a]	Volume, ml.	Temperature	Time, hours	Result	Figure
Formic	C	2	23° [b]	2	Fine dendritic crystals	1
	B	2	23°	2	Fine dendritic crystals	2
Acetic	C	2	23°	2	Short, stubby crystals	3
	B	2	23°	2	None[c]	4
Propionic	C	2	23°	2	None	
	B	2	23°	2	None	
	W	0.2	31°	2	None	
Butyric	W	0.2	31°	2	None	
Isobutyric	W	0.2	31°	2	Very fine "cobwebby" crystals	5
Valeric	W	0.2	31°	2	None	
Isovaleric	W	0.2	31°	2	Sturdy, rather grainy crystals	6
Formic + Propionic + Isovaleric	W	0.2	31°	3.5	Sturdy, rather grainy crystals	7
Formic + Propionic	W	0.2	31°	3.5	None	
Valeric + Isovaleric	W	0.2	31°	3.5	None	

Table I. Crystal Formation During Microdiffusion of Acids to Sodium Hydroxide

(continued)

(a) Acid concentrations approximately 2 percent. Solvents: C = cyclohexane, B = benzene, W = water.

(b) 23° is room temperature.

(c) An indefinite wrinkled film forms on the pellets in all cases.

FIGURES

Figure 1. Sodium formate crystals from diffusion of formic acid from cyclohexane. The sodium hydroxide pellet in each figure is approximately 5 mm. in diameter.

Figure 2. Sodium formate crystals from diffusion of formic acid from benzene.

Figure 3. Sodium acetate crystals from diffusion of acetic acid from cyclohexane.

Figure 4. Product of diffusion of acetic acid from benzene.

Figure 5. Sodium isobutyrate crystals from diffusion of isobutyric acid from water.

Figure 6. Sodium isovalerate crystals from diffusion of isovaleric acid from water.

Figure 7. Product of diffusion of a mixture of formic, propionic and isovaleric acids from water. Note resemblance to Figure 6.